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TRANSMITTAL OF APPEAL BRIEF (Small Entity)	Docket No. MSU 4.1-572

In Re Application Of: **Jes Asmussen and Wen-Shin Huang**

Application No.	Filing Date	Examiner	Customer No.	Group Art Unit	Confirmation No.
10/073,710	02/11/2002	Eric B. Fuller	21036	1762	5422

Invention: **PROCESS FOR SYNTHESIZING UNIFORM NANOCRYSTALLINE FILMS**

COMMISSIONER FOR PATENTS:

Transmitted herewith is the Appeal Brief in this application, with respect to the Notice of Appeal filed on:

February 15, 2006

☒ Applicant claims small entity status. See 37 CFR 1.27

The fee for filing this Appeal Brief is: **$250.00**

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Dated: **February 17, 2006**

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I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to "Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450" [37 CFR 1.8(a)] on **February 17, 2006**.
(Date)

Tammi L. Taylor

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cc:

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.	: 10/073,710	Confirmation No. 5422
Applicants	: Jes Asmussen and Wen-Shin Huang	
Title	: PROCESS FOR SYNTHESIZING UNIFORM NANOCRYSTALLINE FILMS	
Filed	: February 11, 2002	
TC/A.U.	: 1762	
Examiner	: Fuller, Eric B.	
Docket No.	: MSU 4.1-572	
Customer No.	: 21036	

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Alexandria, VA 22313-1450

BRIEF UNDER 37 C.F.R. § 41.37

Sir:

This is an appeal from a final rejection in the above entitled application. The claims on appeal are set forth as Claims Appendix. An oral hearing will be requested. Enclosed is the fee due upon filing of the Brief.

(1) Real Party in Interest

The real party in interest is the Board of Trustees operating Michigan State University, East Lansing, Michigan, a constitutional corporation of the State of Michigan, which is the assignee of the above entitled application.

(2) Related Appeals and Interferences

There are no pending related appeals and interferences.

(3) Status of Claims

Claims 1-5 and 8-19 are pending in the application. Claims 6 and 7 have been cancelled. All claims were rejected. No claims have been allowed. Claims 1-5 and 8-19 are on appeal.

(4) Status of Amendments

An amendments subsequent to final rejection was filed January 05, 2006. The amendment was not entered.

(5) Summary of Claimed Subject Matter

The claimed subject matter in independent Claim 1 is a process for depositing a nanocrystalline diamond film with a grain size between 1 and 100 nm on a surface of a substrate, which comprises:

(a) providing a plasma generating apparatus for depositing the diamond film on the substrate from the plasma, including a plasma source employing a radiofrequency, including UHF or microwave, wave coupler means which is metallic and in the shape of a hollow cavity and which is excited in a TM mode of resonance, and wherein an insulated chamber means has a central longitudinal axis in common with the coupler means and is mounted in closely spaced and sealed relationship to an area of the coupler means with an opening from the chamber means at one end; gas supply means for providing a gas which is ionized to form

the plasma in the chamber means, wherein the radiofrequency wave applied to the coupler means creates and maintains the plasma around the central longitudinal axis (A-A) in the chamber means; movable metal plate means in the cavity mounted in the coupler means perpendicular to the central longitudinal axis and movable along the central longitudinal axis towards and away from the chamber means; and a movable probe means connected to and extending inside the coupler means for coupling the radiofrequency waves to the coupler means;

(b) providing the substrate, wherein the surface to be placed in the plasma has been roughened and cleaned; and

(c) providing the substrate in the insulated chamber means on a substrate holder adjacent to the plasma generated in the chamber means, wherein the gas in the chamber means is at a pressure between 50 and 300 Torr in the presence of the radiofrequency waves for generating the plasma, wherein the gas is ninety percent by volume or more of argon along with methane and optionally hydrogen and essentially free from oxygen or nitrogen and wherein the chamber means is essentially free from leaks of nitrogen or

oxygen or mixtures thereof into the chamber means, so as to generate the plasma and to deposit the nanocrystalline diamond film on the substrate.

Support for this claim is found in original Claim 1 and page 3, line 18 to page 5, line 2 of the specification. Support for a plasma generating apparatus is found described at page 12, line 3 through page 19, line 17 of the specification and illustrated in Figures 1-1G. Support for a substrate, wherein the surface to be placed in the plasma has been roughened and cleaned is found in the Examples under "Seeding Procedure". Support for providing the substrate and depositing the nanocrystalline diamond film on the substrate is found in Example 1.

(6) Grounds of Rejection to Be Reviewed on Appeal

(a) Claims 1-5, 8-12, 14-17 and 19 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 5,311,103).

(b) Claims 13 and 18 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 5,311,103) and further in view of Herb *et al.* (U.S. Patent No. 5,273,790).

(c) Claims 1-5, 8-12, 14-17 and 19 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 4,585,668).

(d) Claims 13 and 18 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 4,585,668) and further in view of Herb *et al.* (U.S. Patent No. 5,273,790).

(e) Claims 1-5, 8-12, 14-17 and 19 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 4,906,900).

(f) Claims 13 and 18 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 4,906,900) and further in view of Herb *et al.* (U.S. Patent No. 5,273,790).

(g) Claims 1-5, 8-12, 14-17 and 19 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 4,727,293).

(h) Claims 13 and 18 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 4,727,293) and further in view of Herb *et al.* (U.S. Patent No. 5,273,790).

(i) Claims 1-5, 8-12, 14-17 and 19 were rejected under the judicially created doctrine of obviousness-type double patenting as being unpatentable over claims 1, 2 and 4 of U.S. Patent No. 4,585,668 in view of Gruen *et al.* (U.S. Patent No. 6,592,839).

(j) Claims 1-5, 8-12, 14-17 and 19 were rejected under the judicially created doctrine of obviousness-type double patenting as being unpatentable over claims 22-27 of U.S. Patent No. 4,585,668 in view of Gruen *et al.* (U.S. Patent No. 6,592,839).

(7) Argument

(a) Claims 1-5, 8-12, 14-17 and 19 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 5,311,103).

According to MPEP §2143, to establish *prima facie* obviousness of a claimed invention, all the claim limitations must be taught or suggested by the prior art. *In re Royka*, 490 F.2d 981, 180 USPQ 580 (CCPA 1974). "All words in a claim must be considered in judging the patentability of that claim against the prior art." *In re Wilson*, 424 F.2d 1382, 1385, 165 USPQ 494, 496 (CCPA 1970).

Gruen *et al.* describes the preparation of nanocrystalline diamond. It is stated in the rejection that Gruen *et al.* explicitly teaches to exclude oxygen and other gases, citing column 4, lines 15-20. However, the cited text refers only to the use of hydrocarbons which consists fundamentally of carbon and hydrogen without added oxygen, chlorine or other elements. Thus, the hydrocarbon used as the carbon source does not have oxygen or other elements as part of its structure, however this statement does not speak

to the exclusion of oxygen or nitrogen gases in the mixture. None of the cited references teach using less than 10 ppm oxygen or nitrogen.

A *prima facie* case of obviousness may be rebutted by showing that the art, in any material respect, teaches away from the claimed invention. *In re Geisler*, 116 F.3d 1465, 1471, 43 USPQ2d 1362, 1366 (Fed. Cir. 1997). This reference teaches away from the present invention, since Applicants teach the importance of excluding nitrogen entirely. While it is true that Gruen *et al.* teaches the use of argon gas, Gruen *et al.* does not explicitly teach the importance of excluding trace amounts of nitrogen from the argon gas. Gruen *et al.* actually positively teach the use of nitrogen as one alternative "inert gas" which can be used. At column 4, lines 24 to 32 Gruen *et al.* states as follows:

> "Similarly, the third component of the plasma in the subject invention is an inert gas. For purposes of simplicity, argon has been used as the inert gas but it is understood by those of ordinary skill in the art that any of the noble gases as well as nitrogen or any mixtures of the noble gases and nitrogen may be used in lieu of argon as an inert gas. Where argon is used in the specification hereafter, it is for purposes of illustration only and not for purposes of limitation."

Thus it is clear that Gruen *et al.* were not concerned about

the presence of nitrogen. According to the MPEP, the totality of the prior art must be considered, and proceeding contrary to accepted wisdom in the art is evidence of nonobviousness. *In re Hedges*, 783 F.2d 1038, 228 USPQ 685 (Fed. Cir. 1986) (Applicant's claimed process for sulfonating diphenyl sulfone at a temperature above 127°C was contrary to accepted wisdom because the prior art as a whole suggested using lower temperatures for optimum results as evidenced by charring, decomposition, or reduced yields at higher temperatures.). Applicant's clearly proceed contrary to the accepted wisdom that nitrogen can be used as one of the gases for the diamond deposition as evidenced by Gruen *et al*.

According to MPEP 2144.05, applicants can rebut a presumption of obviousness based on a claimed invention that falls within a prior art range by showing "(1) [t]hat the prior art taught away from the claimed invention... or (2) that there are new and unexpected results relative to the prior art." *Iron Grip Barbell Co., Inc. v. USA Sports, Inc.*, 392 F.3d 1317, 1322, 73 USPQ2d 1225, 1228 (Fed. Cir. 2004). Applicants can also rebut a *prima facie* case of obviousness based on overlapping ranges by showing the

criticality of the claimed range. "The law is replete with cases in which the difference between the claimed invention and the prior art is some range or other variable within the claims… In such a situation, the applicant must show that the particular range is critical, generally by showing that the claimed range achieves unexpected results relative to the prior art range." *In re Woodruff*, 919 F.2d 1575, 16 USPQ2d 1934 (Fed. Cir. 1990).

Nanocrystallinity and smooth films require high purity deposition conditions, *i.e.* impurities like nitrogen and/or oxygen decrease the film uniformity (Lines 10-12 on page 29 of the specification). Growth rates increase considerably with no hydrogen when pressure is increased up to about 150 Torr (Figure 8). It can be seen in Figures 8A, however that roughness increases above about 120 Torr with no hydrogen. Growth rates increase more dramatically with increasing pressure when 4 sccm hydrogen is present (Figure 9). However, as seen in Figure 9A, surface roughness begins to increase at about 100 Torr. Clearly, surface roughness increases as H_2 concentrations increase and/or pressures increase above pressures in the 100-150 Torr range.

Gruen *et al.* shows in Figure 11 that as the

argon:hydrogen volume percentage increases that the growth rate (μm/hour) decreases. This trend is also taught by Applicants as illustrated in Figure 10 of the specification. Applicants show that the film growth rate increased dramatically with small increases in H_2. The surface RMS roughness (nm) increases when H_2 is increased, as can be seen in Figure 10A. Applicants teach that trace levels of nitrogen interfere with the growth rate (Figure 11), but removing nitrogen does not substantially increase surface roughness (Figure 11A). The growth rates decreased as N_2 impurity increases in the range between 5-2500 ppm (page 30, lines 1-2 of the specification). As shown in Figures 7A-7F and Figure 11, decreasing levels of nitrogen impurity actually increases the growth rate from 0.147 μm/hour at 2500 ppm nitrogen to 0.237 μm/hour at 5 ppm nitrogen. However, decreasing levels of nitrogen impurity only increases the surface roughness from 17.7 nm at 2500 ppm nitrogen to 24.3 nm at 5 ppm nitrogen. This increase in growth rate without substantial increase in surface roughness is not shown or suggested by the prior art references, taken alone or in combination.

In regards to Asmussen, this reference teaches an

apparatus which is not disclosed to be "essentially free of leaks" as set forth in Claim 1 from which each of the other claims depend. One skilled in the art could not derive the claimed invention from the combination of these references.

(b) Claims 13 and 18 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 5,311,103) and further in view of Herb *et al.* (U.S. Patent No. 5,273,790).

Claims 13 and 18 were rejected over the references previously applied in view of Herb *et al.* (U.S. Patent No. 5,273,790). As set forth in Herb *et al.*, molybdenum is known as a substrate holder but not in the process as set forth in Claim 1. None of the references describe coating a silicon carbide seal as in Claim 18 and Figures 18 and 19 (Page 27 of the specification).

(c) Claims 1-5, 8-12, 14-17 and 19 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.*

(U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 4,585,668). Claims 1-5, 8-12, 14-17 and 19 were rejected over Gruen *et al.* in view of Asmussen *et al.* (U.S. Patent No. 4,585,668). Gruen *et al.* has also been discussed previously. Asmussen *et al.* '668 does not disclose an apparatus which was enabled to perform the process as claimed as set forth in connection with Asmussen '103.

(d) Claims 13 and 18 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 4,585,668) and further in view of Herb *et al.* (U.S. Patent No. 5,273,790). Claims 13 and 18 were rejected over Gruen *et al.* in view of Asmussen (U.S. Patent No. 4,585,668) as applied to Claims 1 and 2 in view of Herb *et al.*. As discussed previously in connection with Claims 13 and 18, a molybdenum holder has not been disclosed or suggested in a process as claimed.

(e) Claims 1-5, 8-12, 14-17 and 19 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 4,906,900). Claims 1 to 5, 8 to 12, 14 to 17 and 19 were rejected as unpatentable over Gruen *et al.* in view of Asmussen (U.S. Patent No. 4,906,900). These claims are patentable for the reasons already discussed in relation to Gruen *et al.* and Asmussen '103.

(f) Claims 13 and 18 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 4,906,900) and further in view of Herb *et al.* (U.S. Patent No. 5,273,790). Claims 13 and 18 were rejected over the references previously applied in view of Herb *et al.*. These claims are patentable for the same reasons already discussed.

(g) Claims 1-5, 8-12, 14-17 and 19 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.*

(U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 4,727,293). Claims 1 to 5, 8 to 12, 14 to 17 and 19 were rejected over Gruen *et al.* in view of Asmussen *et al.* (U.S. Patent No. 4,727,293). These claims are patentable for the reasons already discussed in relation to Gruen *et al.*

(h) Claims 13 and 18 were rejected under 35 U.S.C. §103(a) as being unpatentable over Gruen *et al.* (U.S. Patent No. 6,592,839) in view of Asmussen *et al.* (U.S. Patent No. 4,727,293) and further in view of Herb *et al.* (U.S. Patent No. 5,273,790). Claims 13 and 18 were rejected over the references previously applied in view of Herb *et al.* (U.S. Patent No. 5,273,790). These claims are patentable for the reasons previously discussed.

(i) Claims 1-5, 8-12, 14-17 and 19 were rejected under the judicially created doctrine of obviousness-type double patenting as being unpatentable over claims 1, 2 and 4 of U.S. Patent No. 4,585,668 in view of Gruen *et al.* (U.S.

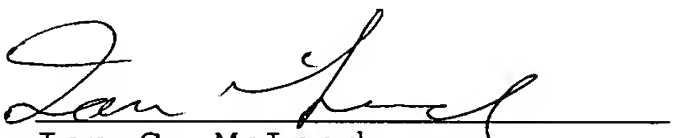
Patent No. 6,592,839). Claims 1 to 5, 8 to 12, 14 to 17 and 19 were rejected on double patenting as being unpatentable over Asmussen U.S. Patent No. 4,585,668 in view of Gruen *et al.*. The presently pending invention represents an improvement not disclosed or suggested or claimed in these references.

(j) Claims 1-5, 8-12, 14-17 and 19 were rejected under the judicially created doctrine of obviousness-type double patenting as being unpatentable over claims 22-27 of U.S. Patent No. 4,585,668 in view of Gruen *et al.* (U.S. Patent No. 6,592,839). Claims 1 to 5, 8 to 12, 14 to 17 and 19 were rejected on double patenting as being unpatentable over Claims 22 to 27 of Asmussen U.S. Patent No. 4,585,668 in view of Gruen *et al.*. These references do not disclose or suggest the improvement claimed.

(b) Conclusion

As shown above, the claimed invention is not obvious over the cited references. Therefore, Claims 1-5 and 8-19 are each patentable. Reversal of the Final Rejection is requested.

Respectfully,



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CLAIMS APPENDIX

1. A process for depositing a nanocrystalline diamond film with a grain size between 1 and 100 nm on a surface of a substrate, which comprises:

(a) providing a plasma generating apparatus for depositing the diamond film on the substrate from the plasma, including a plasma source employing a radiofrequency, including UHF or microwave, wave coupler means which is metallic and in the shape of a hollow cavity and which is excited in a TM mode of resonance, and wherein an insulated chamber means has a central longitudinal axis in common with the coupler means and is mounted in closely spaced and sealed relationship to an area of the coupler means with an opening from the chamber means at one end; gas supply means for providing a gas which is ionized to form the plasma in the chamber means, wherein the radiofrequency wave applied to the coupler means creates and maintains the plasma around the central longitudinal axis (A-A) in the chamber means; movable metal plate means in the cavity mounted in the coupler means perpendicular to the central longitudinal axis and movable along the central longitudinal

axis towards and away from the chamber means; and a movable probe means connected to and extending inside the coupler means for coupling the radiofrequency waves to the coupler means;

(b) providing the substrate, wherein the surface to be placed in the plasma has been roughened and cleaned; and

(c) providing the substrate in the insulated chamber means on a substrate holder adjacent to the plasma generated in the chamber means, wherein the gas in the chamber means is at a pressure between 50 and 300 Torr in the presence of the radiofrequency waves for generating the plasma, wherein the gas is ninety percent by volume or more of argon along with methane and optionally hydrogen and essentially free from oxygen or nitrogen and wherein the chamber means is essentially free from leaks of nitrogen or oxygen or mixtures thereof into the chamber means, so as to generate the plasma and to deposit the nanocrystalline diamond film on the substrate.

2. The process of Claim 1 wherein the substrate has a dimension with a surface area greater than about 20 cm^2.

3. The process of Claim 1 or 2 wherein the microwave is at 2.45 GHz.

4. The process of Claim 1 or 2 wherein the microwave is at 915 MHz.

5. The process of Claim 1 or 2 wherein the film has a thickness of at least about 50 nm micrometers.

8. The process of Claim 1 wherein the substrate is allowed to thermally float at a temperature between about 575°C and 900°C on a side exposed to the plasma.

9. The process of Claim 1 wherein diamond particles

are used for providing the roughened surface by abrasion and wherein the diamond particles have a grain size between about 0.1 to several micrometers, which surface is then cleaned.

10. The process of Claim 1 wherein the pressure on the gas is between about 60 and 240 Torr and at a flow rate of between about 50 and 200 sccm.

11. The process of Claims 1 or 2 wherein the probe means is elongate and is mounted in the coupler means along the central longitudinal axis of the chamber means and coupler means with an end of the probe means in spaced relationship to the chamber means; and wherein stage means in the opening of the chamber which forms part of the cavity and provides for mounting the substrate, the stage means having a support surface which is in a plane around the longitudinal axis and which is pre-adjusted towards and away from the plasma in the chamber means so that the substrate can be coated with the diamond film from the plasma.

12. The process of Claims 1 or 2 wherein the insulated chamber is evacuated so that there is less than about 10 ppm of combined oxygen and nitrogen or nitrogen or oxygen alone as the gas which generates the plasma is provided in the chamber.

13. The process of Claims 1 or 2 wherein the substrate is silicon and wherein the substrate holder is molybdenum.

14. The process of any one of Claims 1 or 2 wherein the substrate on which the diamond is deposited has a surface area with a diameter which is greater than about 8 cm.

15. The process of Claim 1 wherein the gas contains about 1% methane.

16. The process of Claim 1 wherein the mode of the plasma is selected from the group consisting of TM012 and TM013.

17. The process of Claim 1 wherein at pressures of greater than about 250 Torr the stage means can be optionally cooled.

18. The process of Claim 1 wherein the substrate is a silicon carbide seal and the holder is molybdenum which shields a first portion of the seal while allowing a portion of the seal to be coated with the nanocrystalline diamond.

19. The process of Claim 1 wherein the apparatus has a static magnetic field around the plasma which aids in coupling radiofrequency energy at electron cyclotron resonance and aids in confining ions in the plasma in an electrically insulated chamber means in the coupler means.